PneumatiCoat TECHNOLOGIES

Commercializing "nano"

Scale-up of Low-Cost Encapsulation Technologies for High Capacity and High Voltage Electrode Powders

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www.pneumaticoat.com

Project ID: ES239

Overview

Timeline (Phase I & II)

- June 10, 2013
- July 26, 2016
- Phase II is 93% Complete

Budget

- Total SBIR Project Funding
  - 100% by DOE

Barriers

- Poor stability of high energy density materials
- Perceived cost of stabilizing coatings
- Overbuilding requirements to meet cycle life targets

Collaborators

- Nader Hagh, NEI Corp.
- Sung-Jin Cho, NC A&T Univ.
- Fabio Albano, XALT Energy LLC
- Corporate Partnerships

Main Objectives

PHASE I

- ✓ Compare performance of stabilizing approaches: ALD vs. co-precipitation
- ✓ Demonstrate low-cost ALD using an innovative semi-continuous manufacturing approach
- ✓ Validate economic viability of semi-continuous ALD
- ✓ Develop new ALD coating chemistries for high capacity (HC) and high voltage (HV) materials

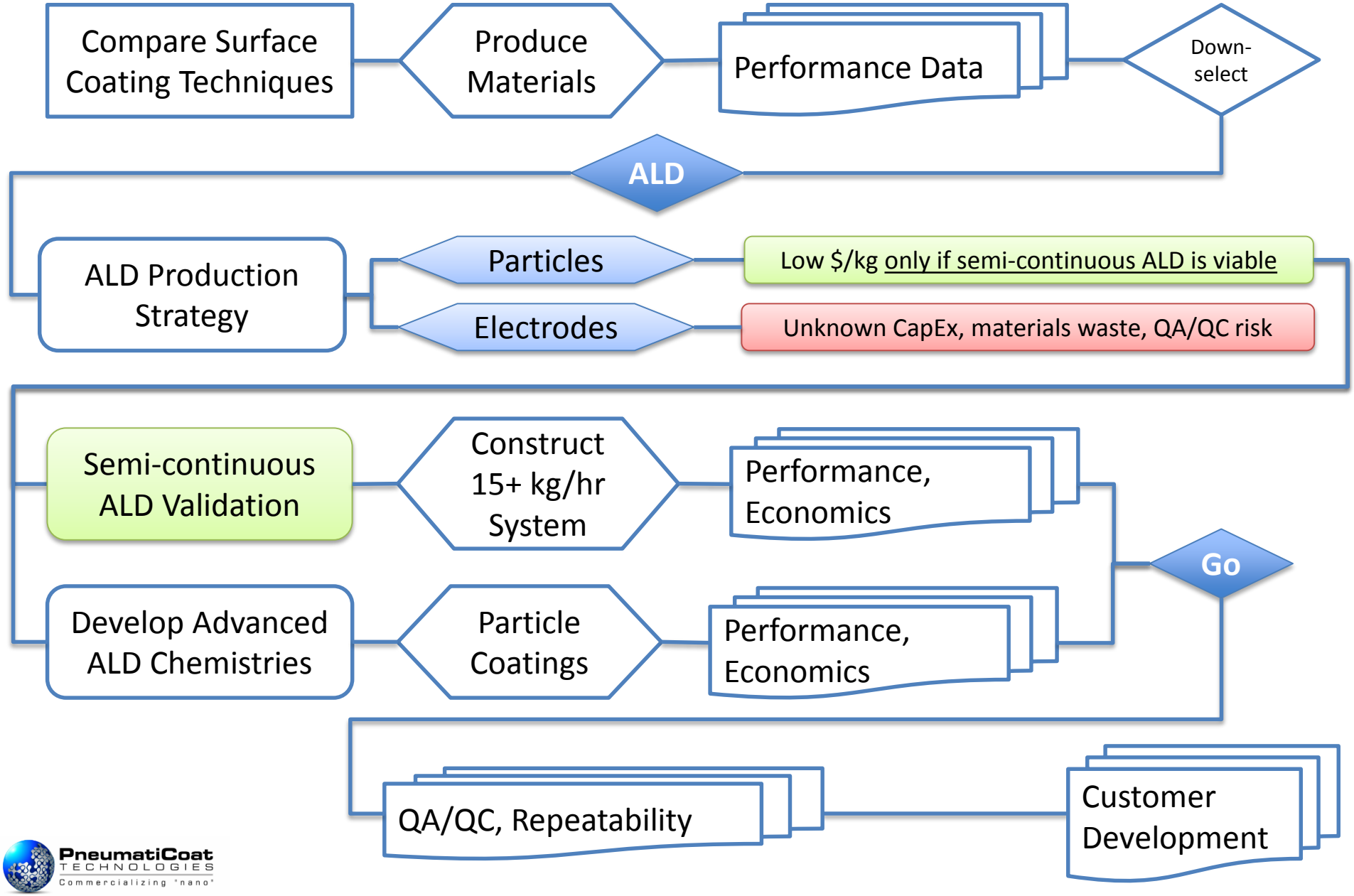
PHASE II

- ✓ Construct and validate a 100 kg/shift Particle ALD reactor
- ✓ Demonstrate and down-select new ALD coating chemistries for HV cathodes
- ✓ Implement QA/QC strategy for ALD manufacturing
- ✓ Produce over 400 kg of material for strategic partners
- ✓ Demonstrate < 5% capacity fade over 200 cycles in 2+ Ah pouch and 18650 cells

Phase II Milestones

Milestone # and Description	Milestone Verification Process	Month
M1: Pilot Reactor Installation	Successful system construction and ability to control critical process variables such as pressure, temperature and valve firing in automatic operation	5 **Complete**
M2: 2Ah Baseline Cells	Successful fabrication of viable 2Ah cells and completion of 200 testing cycles at 1C rates	6 **Complete**
M3: Pilot Reactor Commissioning	Completed recipe builds for Al_2O_3 and TiO_2 ALD processes yielding > 80% material collection and < 5% variation in coating content per cycle	11 **Complete**
M4: Year 1 Report	Successful completion of Tasks 1-1 to 1-8	12 **Complete**
M5: Final Down-Selection	Successful identification of the coating processes for LNMO and graphite powders providing the greatest value proposition.	18 **Complete**
M6: Year 2 Report, Phase III	Successful completion of Tasks 2-1 to 2-6	24

Technical and Strategic Approach



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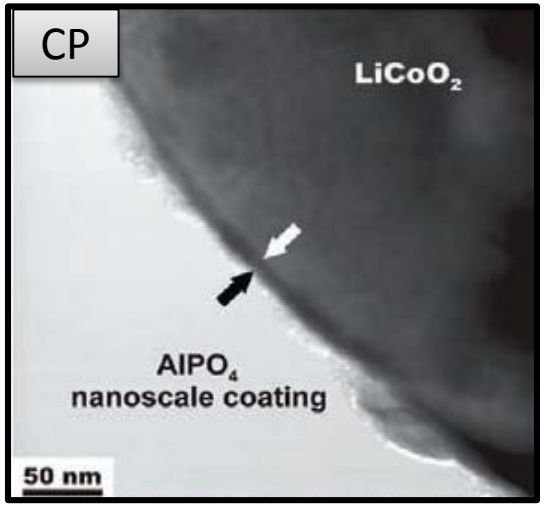
Technical and Strategic Approach

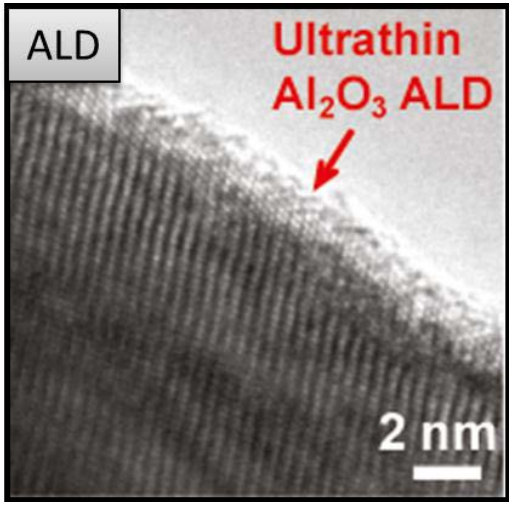
Balanced value proposition assessment between gas-phase and liquid-phase surface coating techniques: *Co-Precipitation (CP) versus ALD*

Select Particles:
HV LNMO,
HC NMC (layered)

Select Coatings:
Al_2O_3, TiO_2



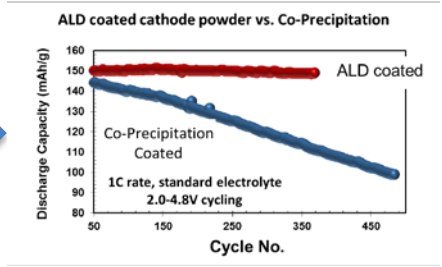
K.T. Lee, et al.



I.D. Scott, et al

CP → NEI Corporation

ALD → PneumatiCoat TECHNOLOGIES Commercializing "nano"



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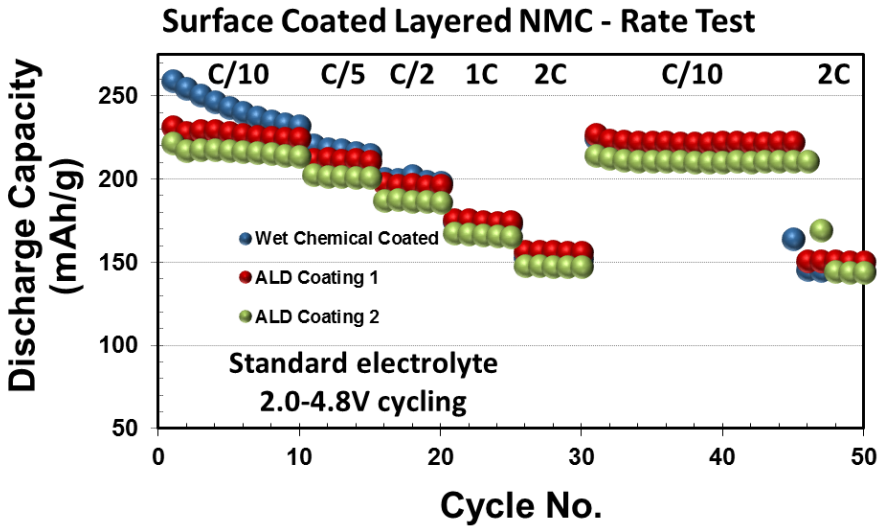
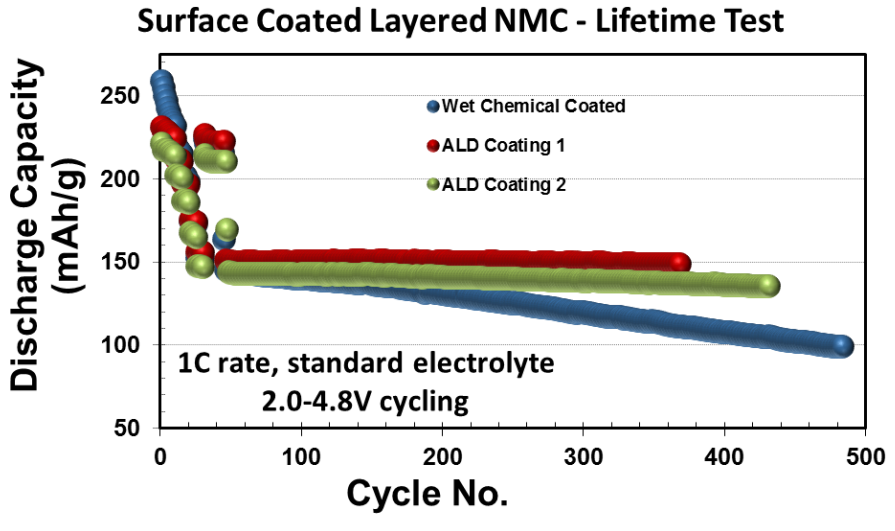
K.T. Lee, et al., "Roles of Surface Chemistry on Safety and Electrochemistry in Lithium Ion Batteries" Accounts Chem. Res., DOI:10.1021/ar200224h

I.D. Scott, et al., "Ultrathin Coatings on Nano-$LiCoO_2$ for Li-Ion Vehicular Applications" *Nano Letters,* 2011. 11(2): p. 414-418.

ALD vs. CP for HV and HC Cathode Powders



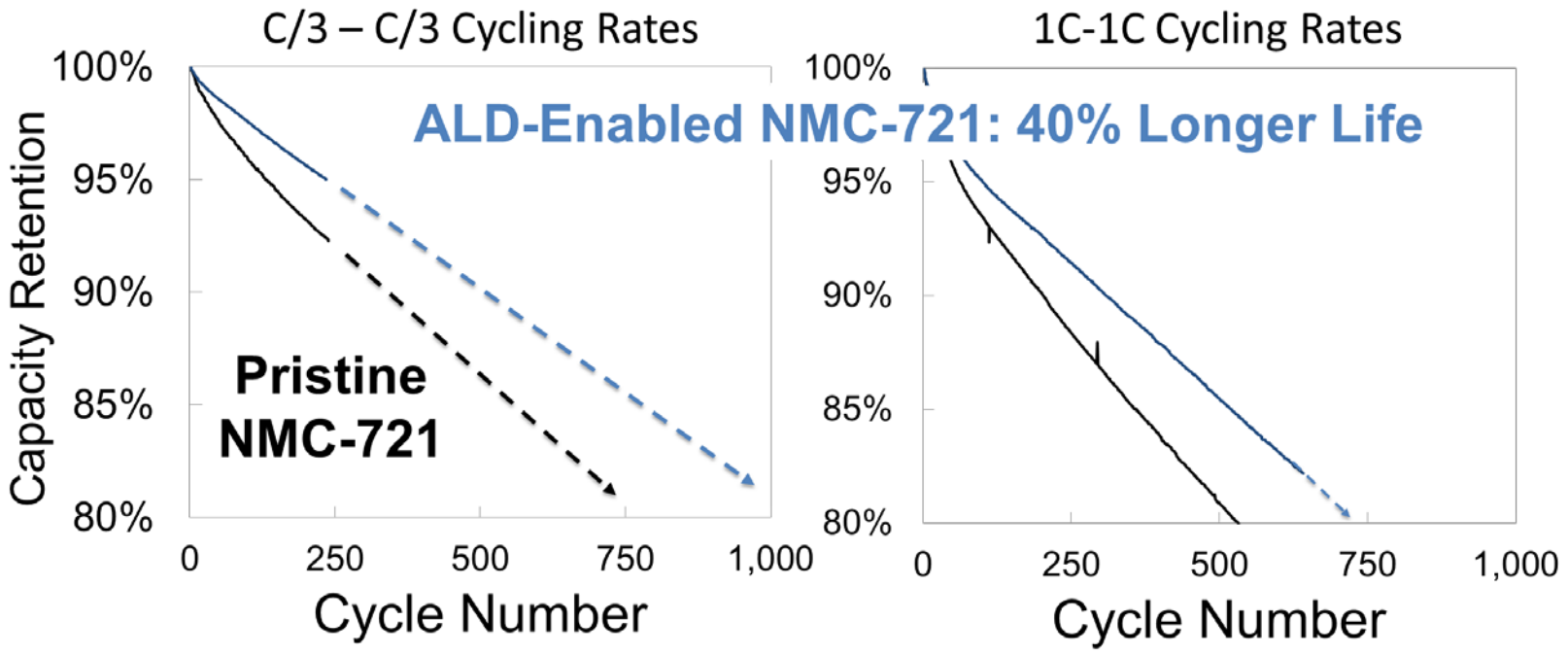


- Initially, CP coatings (2-4 wt%) maintained high performance, but were not robust and failed during long-term testing
- ALD coatings (0.1-0.5 wt%) demonstrated robust performance over long-duration cycling for both 5 V LNMO and layered LNMC

- Decision Point: GO for ALD coatings based on more robust performance and lower materials consumption
- Optimal ALD thickness and materials down-selected for each

Pouch Cell ALD Performance Validation

2 Ah pouch cells fabricated by XALT Energy using PCT's ALD-enabled NMC-721 and Pristine Graphite



Demonstrated performance: 190 Wh/kg, 430 Wh/L, 1000 cycles at C/3 rate

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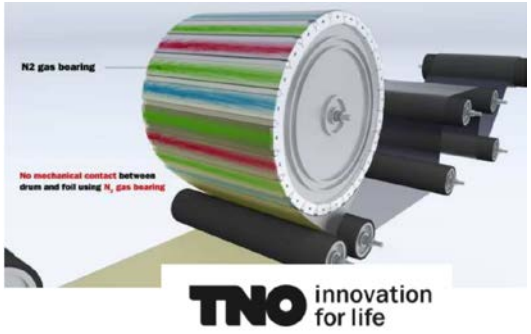
XALT® Energy

ALD Production Strategy: Particles vs. Electrodes

- ALD on electrodes requires Roll-to-Roll processing
  - Highest likelihood of success: TNO / Holst Centre efforts
  - *However precursor waste is 50-70% (Holst)*
  - ALD/MLD-coated electrodes benefit from diffusion time, particularly for thick electrodes: "slow" R2R is anticipated, increasing $/m^2
  - Process risk: failure would scrap embedded cost of finished electrode
  - Still many unanswered questions for large global investment to date
  - Customer development efforts suggest value chain is unwilling to bear the high risk of adopting an ALD-coated electrode approach



- ALD on particles requires <u>high-throughput</u> processing
  - Batch Fluidized Bed Reactor ALD: expensive and not scalable
  - <u>Semi-continuous ALD: low-cost, focus of this program</u>
  - Continuous ALD: infeasible due to costs, entrainment losses

If Successful, Semi-continuous Particle ALD Coating Provides Lowest $/kWh

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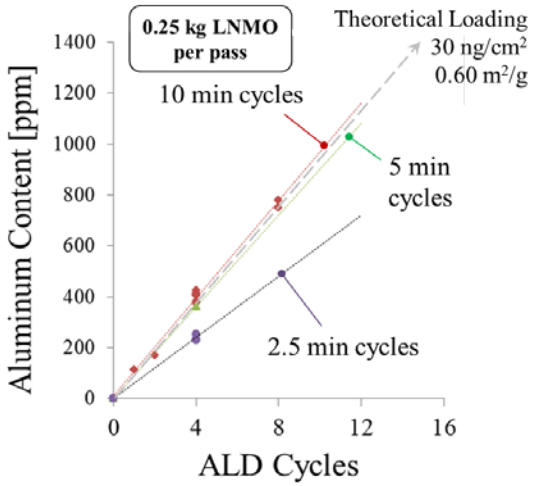
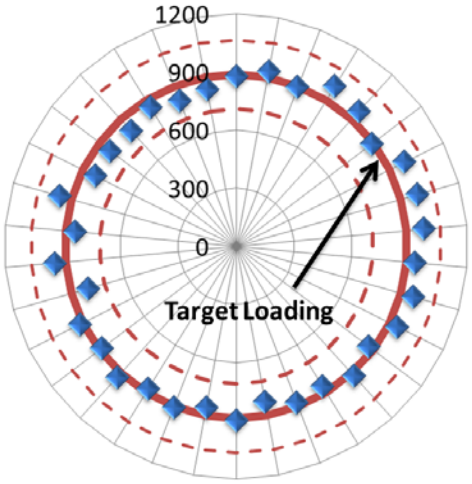
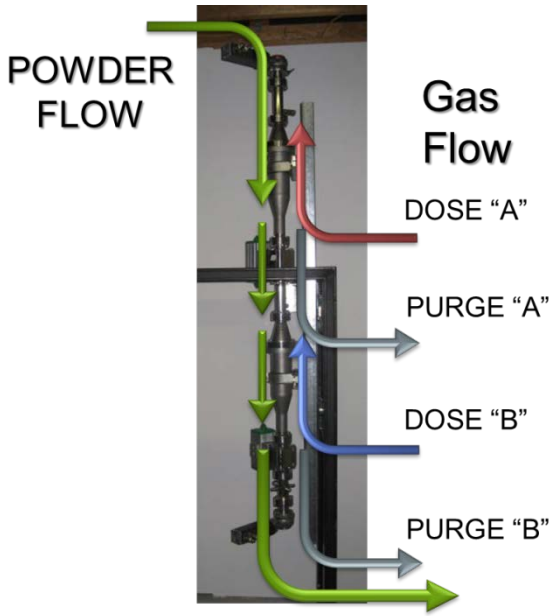
Semi-Continuous Particle ALD Validation

Phase I Prototype Scale (3 kg/hr)







- Linear ALD growth with cycles achieved
- Adds residence time as variable
- Minimizes water exposure for sensitive electrode powders
- Low CapEx and utilities costs; 200x throughput over batch systems
- Fully automated using conventional powder handling equipment
- Makes powder flow, not precursor flow, rate determining step

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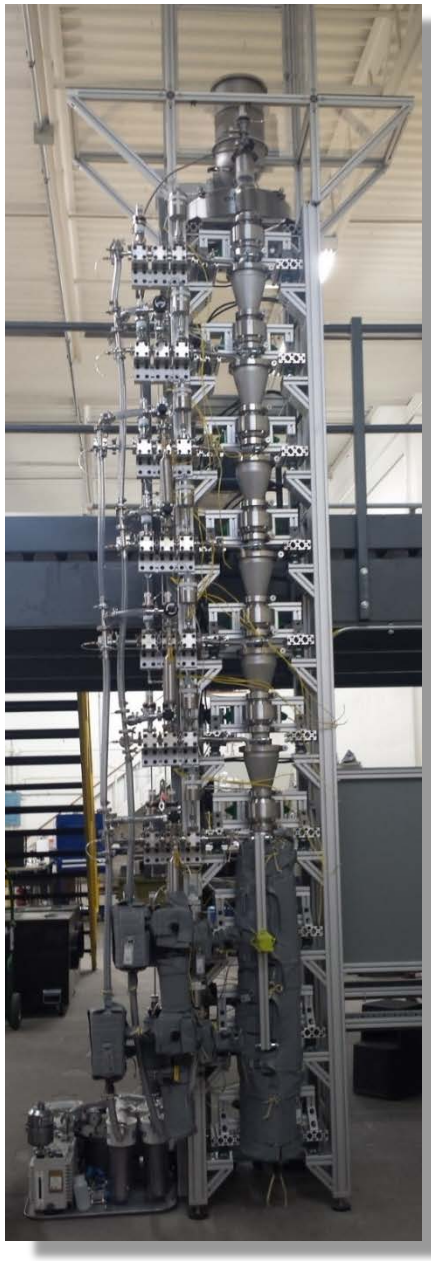
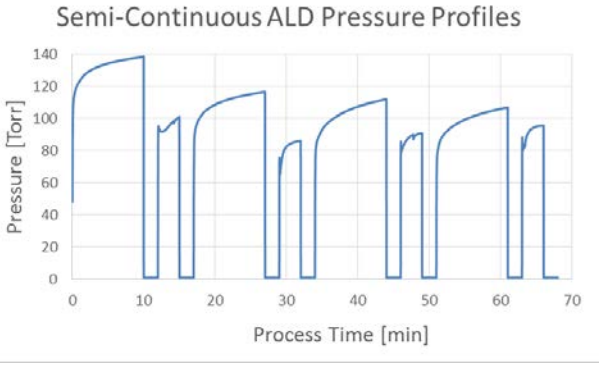
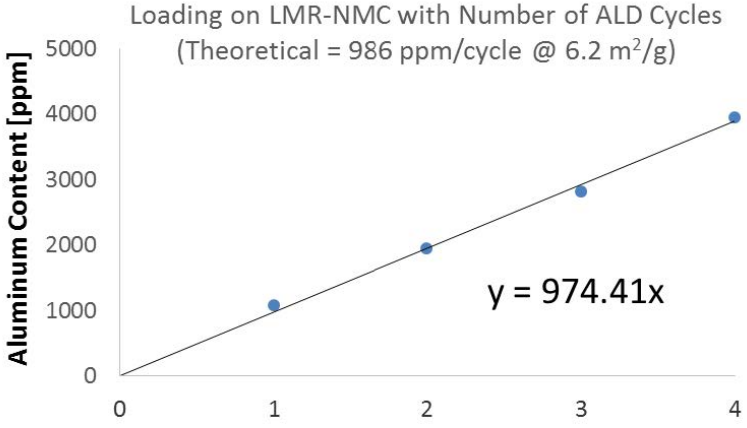
Phase II: Validation of Pilot-Scale Manufacturing

- 200 kg/day 4-ALD cycle system
- Produced > 1,700 kg in prev. 12 mo.
- Linear growth with cycles
- Fully-automated controls
- Currently designing 1 MT/day system for 3Q16 commissioning







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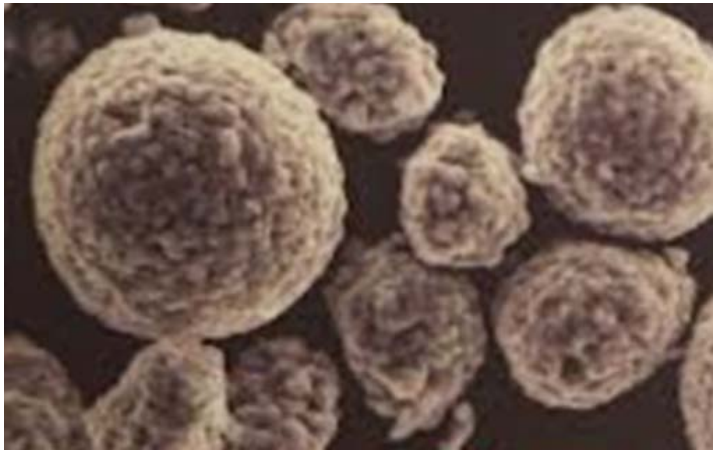
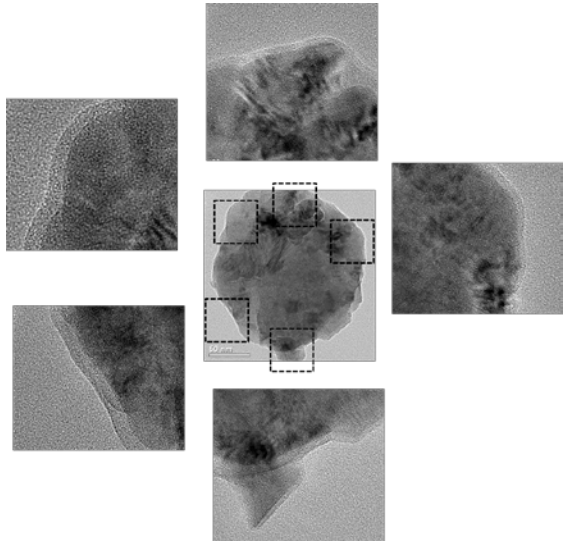
Phase II: Validation of Pilot-Scale Manufacturing

- Suitable for coating entire surface area of Li-ion battery powders, which consist of agglomerates of primary powders
- PCT's alternate process sequence yields better performance than "traditional" ALD process





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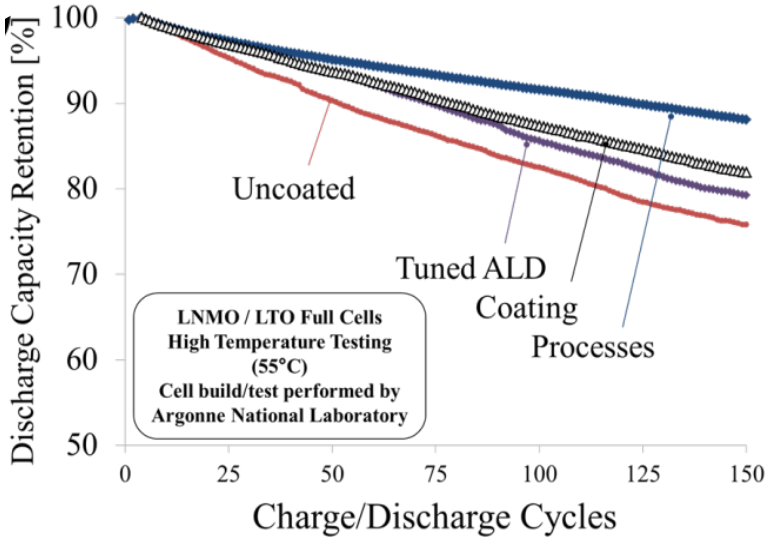
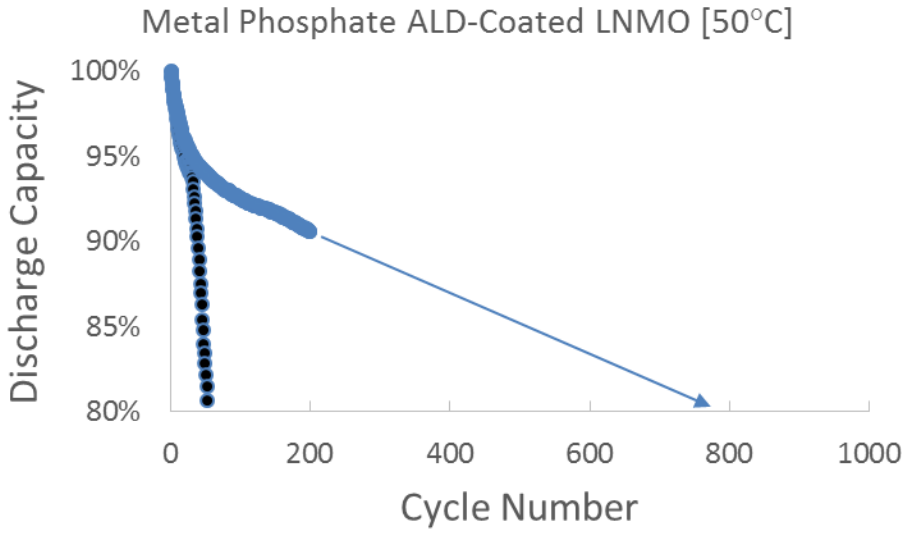
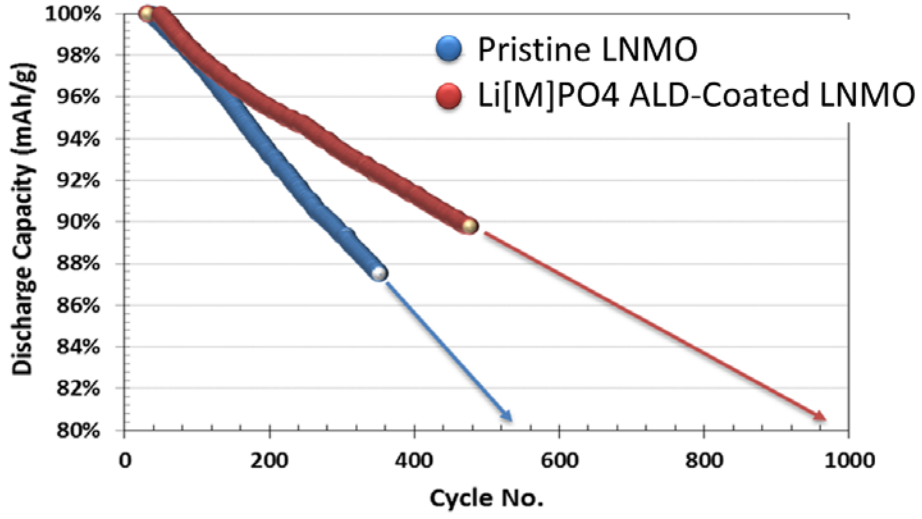
Alternate ALD Chemistry Development: Li[M]PO_4

ALD-Oxide LNMO/LTO EOL performance: 300 cycles at 55°C





Transitioning to [M]PO_4 and Li[M]PO_4 ALD Processes significantly enhances ALD-enabled LNMO



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PneumatiCoat SBIR Program Summary

- ✓ Confirmed ALD delivers highest value proposition of all surface coating techniques
- ✓ Validated scalability of Particle ALD using semi-continuous manufacturing approach
  - ✓ Pilot-scale system capable of 200 kg/day
  - ✓ Light-commercial scale systems under design
  - ✓ Fewer unanswered questions vs. ALD electrode coating
- ✓ Developed ALD processes for fluorides, phosphates, Li-containing coatings, and others
  - ✓ No-Go for fluorides: hygroscopic, waned commercial interest
  - ✓ Li-containing coatings provide 20% reduction in 1st cycle ICL

- ✓ Issued Patent: US 8,993,051 for ALD-enabled battery materials
- ✓ Issued Patent: US 9,284,643 for High Throughput ALD Apparatus